



## INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

<p>(51) International Patent Classification <sup>5</sup> : D01F 2/00, D01D 5/06, 5/088</p>	<p>A1</p>	<p>(11) International Publication Number: WO 94/28218 (43) International Publication Date: 8 December 1994 (08.12.94)</p>
<p>(21) International Application Number: PCT/GB94/01107 (22) International Filing Date: 20 May 1994 (20.05.94) (30) Priority Data: 08/066,522 24 May 1993 (24.05.93) US (71) Applicant: COURTAULDS FIBRES (HOLDINGS) LIMITED [GB/GB]; 50 George Street, London W1A 2BB (GB). (72) Inventors: WHITE, Patrick, Arthur; 51 Park View, Sharnford, Leicestershire LE10 3PT (GB). HAYHURST, Malcolm, John; 90 Bridgeacre Gardens, Coventry CV3 2NP (GB). OWENS, Alan; 22 Newlyn Close, Horeston Grange, Nuneaton CV11 6GG (GB). ROUGHSEGE, Ian, David; 4950 Government Boulevard 75, Mobile, AL 36693 (US). DAVIES, Richard, James; 8 St. Francis Grove, Laceby, Grimsby, South Humberside DN37 7HG (GB). SELLARS, Alan; 2 Trinity Close, Goxhill, South Humberside DN19 7NN (GB). MACDONALD, Jacqueline, Faye; 26 Brierley Road, Henley Green, Coventry CV2 1RS (GB). QUIGLEY, Michael, Colin; 41 Bonneville Close, Meriden CV5 9QH (GB). DRAPER, Ralph; 103 Nunts Lane, Coventry CV6 4GZ (GB).</p>	<p>(74) Agent: NEWBY, John, Ross; J.Y. &amp; G.W. Johnson, Furnival House, 14-18 High Holborn, London, WC1V 6DE (GB). (81) Designated States: AT, AT (Utility model), AU, BB, BG, BR, BY, CA, CH, CN, CZ, DE, DE (Utility model), DK, ES, FI, GB, HU, JP, KP, KR, KZ, LK, LU, LV, MG, MN, MW, NL, NO, NZ, PL, PT, RO, RU, SD, SE, SI, SK, TT, UA, UZ, VN, European patent (AT, BE, CH, DE, DK, ES, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR, NE, SN, TD, TG).  Published <i>With international search report. Before the expiration of the time limit for amending the claims and to be republished in the event of the receipt of amendments.</i></p>	
<p>(54) Title: SPINNING CELL</p> <p>(57) Abstract</p> <p>A method of producing lyocell fibres (125) by spinning a solution of cellulose in an organic solvent through an air gap and into a spin bath (115) in which there is provided means (121, 122) to generate a cross-draught of air in the air gap.</p>		

**FOR THE PURPOSES OF INFORMATION ONLY**

Codes used to identify States party to the PCT on the front pages of pamphlets publishing international applications under the PCT.

AT	Austria	GB	United Kingdom	MR	Mauritania
AU	Australia	GE	Georgia	MW	Malawi
BB	Barbados	GN	Guinea	NE	Niger
BE	Belgium	GR	Greece	NL	Netherlands
BF	Burkina Faso	HU	Hungary	NO	Norway
BG	Bulgaria	IE	Ireland	NZ	New Zealand
BJ	Benin	IT	Italy	PL	Poland
BR	Brazil	JP	Japan	PT	Portugal
BY	Belarus	KE	Kenya	RO	Romania
CA	Canada	KG	Kyrgyzstan	RU	Russian Federation
CF	Central African Republic	KP	Democratic People's Republic of Korea	SD	Sudan
CG	Congo	KR	Republic of Korea	SE	Sweden
CH	Switzerland	KZ	Kazakhstan	SI	Slovenia
CI	Côte d'Ivoire	LJ	Liechtenstein	SK	Slovakia
CM	Cameroon	LK	Sri Lanka	SN	Senegal
CN	China	LU	Luxembourg	TD	Chad
CS	Czechoslovakia	LV	Latvia	TG	Togo
CZ	Czech Republic	MC	Monaco	TJ	Tajikistan
DE	Germany	MD	Republic of Moldova	TT	Trinidad and Tobago
DK	Denmark	MG	Madagascar	UA	Ukraine
ES	Spain	ML	Mali	US	United States of America
FI	Finland	MN	Mongolia	UZ	Uzbekistan
FR	France			VN	Viet Nam
GA	Gabon				

- 1 -

Spinning Cell

This invention relates to spinning cells and has particular reference to spinning cells used for the coagulation of lyocell filaments.

5 As used herein, the term "lyocell" is defined in accordance with the definition agreed by the Bureau International pour la Standardisation de la Rayonne et de Fibres Synthetique (BISFA) namely:-

"A cellulose fibre obtained by an organic solvent  
10 spinning process; it being understood that:-

(1) an "organic solvent" means essentially a mixture of organic chemicals and water; and

(2) "solvent spinning" means dissolving and spinning without the formation of a derivative".

15 Thus a lyocell fibre is produced by the direct dissolution of the cellulose in a water containing organic solvent - typically N-methyl morpholine N-oxide - without the formation of an intermediate compound. After the solution is extruded (spun) the cellulose is precipitated as a fibre.  
20 This production process is different to that of other cellulosic fibres such as viscose, in which the cellulose is first converted into an intermediate compound which is then dissolved in an inorganic "solvent". The solution in the viscose process is extruded and the intermediate compound is  
25 converted back into cellulose.

The general process for the preparation of lyocell fibres is described and illustrated in US Patent 4,416,698, McCorsley, the contents of which are incorporated herein by way of reference.

30 The present invention is particularly concerned with the

- 2 -

spinning cell into which the extruded fibres pass after leaving the spinnerette or jet, first passing through an air gap and then into a coagulation bath.

Accordingly, in one aspect, the present invention  
5 provides a spinning cell for the coagulation of filaments from a solution of cellulose contained in an organic solvent for the cellulose, the cell including a spin bath for the leaching of the solvent from the filaments and a gap above the spin bath, the gap being defined at the lower side by the surface  
10 of the spin bath and at the upper side by a spinnerette from which the filaments emerge, and having means to provide a flow of gas across the gap. The means preferably comprise a suck nozzle having an entrance on one side of the gap.

In another aspect the invention provides a method for the  
15 production of cellulose filaments from a solution of cellulose in an organic solvent, which comprises the steps of extruding the solution through a die having a plurality of holes to form a plurality of strands, passing the strands across a gaseous gap into a water-containing spin bath to form the filaments  
20 and providing a forced flow of gas through the gap parallel to the upper surface of the water in the spin bath by providing a flow of gas across the gap. The gas may be sucked across the gap.

As indicated above, the invention is particularly  
25 suitable for the production of lyocell filaments.

The gap may conveniently be an air gap and a blow nozzle having an exit on one side of the air gap may be provided on the opposite side of the air gap to the suck nozzle.

The suck nozzle preferably has a greater cross-sectional  
30 area at its entrance than the blow nozzle has at its exit.

Baffle means may be located within the spin bath to restrict the flow of currents of liquid within the spin bath

- 3 -

and to calm the surface of the liquid, and in another aspect the invention provides a spinning cell for the coagulation of cellulose filaments formed from a solution of cellulose in an organic solvent, characterised in that the cell has a spin bath for the leaching of the solvent from a tow of the filaments as it is passed through the spin bath, the spin bath having baffles to reduce turbulence.

The invention also provides a method for the production of cellulose filaments from a solution of cellulose in an organic solvent, characterised in that the solution is extruded through a die having a plurality of holes to form a plurality of filaments, the filaments are passed as a tow through a water-containing spin bath to leach solvent from the filaments, and baffles are provided in the spin bath to reduce turbulence.

In a yet further aspect the invention provides a spinning cell for the coagulation of cellulose filaments formed from a solution of cellulose in an organic solvent, characterised in that the cell has a spin bath for the leaching of the solvent from a tow of the filaments, the lower end of the spin bath having a hole through which the tow can be passed, the hole being provided with a resilient periphery to resiliently contact the tow.

The invention also provides a method for the production of cellulose filaments from a solution of cellulose in an organic solvent, characterised in that the solution is extruded through a die having a plurality of holes to form a plurality of filaments, the filaments are passed through a water-containing spin bath to leach solvent from the filaments and the tow of the filaments is passed through a hole at the lower end of the spin bath, the hole being provided with a resilient periphery to resiliently contact the tow.

The resilient periphery may be provided by a cylindrical gaiter of flexible resilient material having an orifice which

- 4 -

in the unrestrained condition is slightly smaller in cross-sectional area than the tow, the gaiter being sealingly secured at its upper end around the aperture in the lower end of the spin bath, the tow passing, in use, through the orifice 5 and thereby expanding the cross-sectional area of the orifice in the gaiter.

The apparatus of the invention may include, as required, means to supply spin bath liquor to a spin bath;

means to remove spin bath liquor from the spin bath; and

10 means to supply air of defined temperature and humidity to the blow nozzle, where provided.

The solvent used to dissolve the cellulose is preferably an aqueous n-methyl morpholine N-oxide solvent.

The temperature of the air in the air gap is preferably 15 maintained below 50°C and above the temperature which would cause freezing of water within the strands of the mixture and the relative humidity of the air is preferably maintained below a dew point of 10°C.

The length of the strands in the gaseous, e.g. air, gap 20 is preferably maintained in the range 0.25 to 50 cm.

The die through which the solution is extruded may have in excess of 500 holes and may have between 500 and 100,000 holes, preferably between 5,000 and 25,000 holes and further 25 preferably between 10,000 and 25,000. The holes may have a diameter in the range 25 microns to 200 microns.

The solution of cellulose may be maintained at a temperature in the range 90°C to 125°C.

As indicated above, the gas may be air and the air may 30 be both sucked and blown across the air gap and the air gap

- 5 -

may have a height between 0.5 cm and 25 cm. The solution may be extruded substantially vertically downward into the spin bath. The air may have a dew point of 10°C or below and may have a temperature in the range 0°C to 50°C.

- 5       The filaments may be extracted from a hole in the bottom of the spin bath, and the hole may be provided with a flexible gaiter to contact the filaments passing therethrough so as to reduce spin bath liquid passage through the hole.

There may be a weir surface to define the upper level of  
10 liquid in the spin bath. The weir may be defined by at least one edge of the spin bath. There may be provided a drainage passage down the side of the spin bath adjacent the weir. There may be a water trap in the drainage passage. The spinning cell may be rectangular in shape with a blow nozzle  
15 on one longer side and the suck nozzle on the opposed longer side. There may be an access door in one or both shorter sides of the cell. The upper edge of the cell on the suck side may act as a weir to define the level of liquid in the cell. There may be a drainage passage on the outside of the  
20 wall having the weir. The drainage passage may include a liquid trap to prevent air being sucked up the passage.

Baffles may be provided at a plurality of levels in the cell. The baffles may comprise apertured plates.

There may be provided a thermally insulating layer  
25 beneath the side walls of the spinnerette on at least the blow side. The insulating layer may be provided on the blow side and on the two short sides.

By way of example embodiments of the present invention will now be described with reference to the accompanying  
30 drawings of which:-

Figure 1 is a cross sectional view along a minor axis of a jet assembly,

- 6 -

Figure 2 is a cross section of a portion of Figure 1 perpendicular to the section of Figure 1,

Figure 3 is a perspective view of a spinnerette,

Figure 4 is an underneath plan view of the spinnerette 5 and insulation,

Figure 5 is a perspective view of one form of spinning cell,

Figure 6 is a perspective view of a second form of spinning cell,

10 Figure 7 is a perspective view of the upper portion of the spinning cell of Figure 6 showing the air gap,

Figure 8 is a cross-sectional view of the exit from the spinning cell,

Figure 9 is a perspective view of the top of a spin bath,  
15 and

Figure 10 is a cross-sectional view of a water trap.

The invention can most clearly be understood by comparisons of the drawings attached hereto with the invention described and illustrated in US Patent 4,416,698.

20 In Figure 2 of UP Patent 4,416,698, it can be seen that the solution of cellulose in amine oxide and non-solvent - typically water - is extruded through a jet or spinnerette 10 to form a series of filaments which pass through an air gap into a water bath. The filaments then pass around a roller  
25 12 to emerge from the upper surface of the water bath. When the filaments emerge from the spinnerette 10 and encounter the air gap they are stretched within the air gap. When the filaments enter the liquid in the spin bath the solvent



- 7 -

leaches out of the filaments to re-form the filaments so as to produce the cellulosic filaments themselves.

The number of filaments produced by the spinnerette in the prior reference US Patent 4,416,698 is low - typically 32 5 filaments are produced, see Example 1 (column 6, line 40).

Although such low numbers of filaments may be suitable for the preparation of filamentary lyocell yarn, when it is required to produce staple fibre, then it is necessary to spin very large numbers of filaments simultaneously. Typically in 10 excess of 5,000 filaments would be produced per spinning cell and a plurality of spinning cells would be arranged in a side-by-side location to produce very large numbers - in the hundreds of thousands - of filaments which could be washed and cut to form staple fibre.

15 The invention provides a spinning cell in which there is provided a cross-draught of air in the air gap to cool the filaments as they emerge from the spinnerette. Typically the temperature at which the cellulose solution is extruded through the spinnerette is in the range 95°C to 125°C. If the 20 temperature drops too low, the viscosity of the cellulose solution becomes so high that it is impractical to extrude it through a spinnerette. Because of the potential exothermic nature of the cellulose solution in N-methyl morpholine N-oxide (herein NMMO), it is preferred that the temperature 25 of the solution - sometimes referred to as a dope - is maintained below 125°C, preferably below 115°C. Thus the temperature of the dope in the spinnerette is close to at or above the boiling point of the water which is typically used in the spin bath. The contents of the spin bath may be water 30 alone or a mixture of water and NMMO. Because the NMMO is continuously leached from the filaments into the spin bath, the spin bath would, during normal operation, always contain NMMO.

The provision of the cross-draught of air in the air gap

- 8 -

has been found to stabilise the filaments as they emerge from the spinnerette, thus enabling larger numbers of filaments to be spun at a given time and enabling the simultaneous production of the large number of filaments required for the manufacture of staple fibre on a commercial scale.

The use of a cross-draught enables the gap between the face of the spinnerette and the liquor in the spin bath to be kept to a minimum level, hence reducing the overall height of the spinning cell.

10 For optimum performance the humidity of the air should be controlled so that it has a dew point of 10°C or less. The dew point may be in the range 4°C to 10°C. The temperature of the air can be in the range 5°C to 30°C, but the air can be at 10°C with a relative humidity of 100%.

15 Referring to Figure 5 this shows a spinning cell 101 which has a generally rectangular shape with a prismatic portion 102 towards the lower end. At the bottom of the cell is an outlet hole 103 which will be described in further detail below. The upper edge 104 of the spinning cell defines  
20 the upper level of liquor in the spinning cell. Typically the liquor contained in the cell would be a mixture of water and 25% NMMO, but concentrations in the range 10% to 40% or 20% to 30% by weight of NMMO can be used. The dotted lines 105, 106, define the path of the filaments passing through the spin  
25 bath during the leaching process. At the upper end of the cell the filaments are in a generally rectangular array 107. The shape of the array 107 will be defined by the shape of the spinnerette or jet through which the filaments are extruded in the spinning process. To prevent excessive turbulence of  
30 spin bath liquor within the cell, perforated plates 108, 109, 110 having 3mm holes and 40% voidage are located within the upper region of the cell to restrict flow of cell liquor within the cell.

As the filaments pass downwardly in a tow through the

- 9 -

cell they entrain spin bath liquor held at 25°C, or in the range 20°C to 30°C and the entrained liquor is carried downwardly. Because the total cross sectional area of the tow of filaments is reduced as they approach the outlet, excess spin bath liquor is expressed sideways from the tow of filaments. This sets up a pumping action of liquor within the bath, tending to produce currents of liquor in the cell. The use of the porous baffles 108, 109 and 110 significantly reduces turbulence of the surface of the spin bath and within the upper portion of the bath. This reduction in turbulence prevents or significantly reduces splashing of the spin bath liquor up onto the face of the spinnerette and prevents disruptive movement of the filaments.

As shown in Figure 6, the baffles 111 and 112 are preferably shaped so as to be quite close to the moving surfaces of the tow or tows of filaments passing downwardly through the cell. In the case of the use of a spinnerette which forms the filaments into two rectangular tows 113, 114 which pass downwardly through the spinning cell as prismatic regions 115, 116 until they combine to emerge through the hole 103 at the bottom of the spinning cell.

Referring to Figure 7, this shows in more detail the air gap and the cross-draught arrangement. The spin bath 115 which has an upper surface 116 defined by the edges 117, 118, 119 and 120 of the spinning cell. Effectively the edges act as dams or weirs and a slight excess of spin bath liquor is passed into the cell to flow over the weirs so as to form a surface 116 of constant location and therefore of fixed height.

A cross-draught in the form of air having a temperature in the range 10°C to 40°C and a relative humidity in the range of dew points 4°C to 10°C is blown across the air gap from a blow nozzle 121 into a suction nozzle 122. Air is sucked through the nozzle 122 so as to maintain a parallel flow of air across the spin bath. The thickness of the blow nozzle

- 10 -

121 is about one quarter to one fifth of the thickness of the suction nozzle 122. The lower edge 123 of the suction nozzle 122 is substantially at the same level as the edge 119 of the spin bath. The edge 123 may be slightly below the level of 5 the spin bath edge 119. Air typically at 20°C is blown at 10 metres/second across the air gap.

Typically the suck nozzle 122 would have a thickness of about 25 mm and the air gap would then be about 18 to 20 mm high.

10 The jet assembly 124 which produces the filaments 125 preferably comprises a spinnerette formed of thin sheets of stainless steel welded into a structure which has a flat under surface mounted in an assembly which provides heat to the spinnerette and which thermally insulates the bottom of the 15 spinnerette. Such spinnerettes are ideally suited to a spinning cell according to the present invention in that the cross-draught of air has been found to stabilise the filaments emerging from the spinnerette.

Referring to Figure 1, this shows a jet assembly located 20 within an insulating cover 1 and frame 2. The frame 2 is thermally insulated from its steel support structure, and has a bore 3 extending around the frame through which a suitable heating medium such as hot water, steam, or oil, can be passed to heat the lower end of the frame. Because the cellulose 25 solution spun through the jet assembly is supplied to the jet assembly at an elevated temperature, typically 105°C, it is preferable to provide heating to maintain the solution at the correct temperature and to provide insulation to minimise excessive heat loss and to prevent injury to operating 30 personnel.

Bolted to the frame 2 by means of bolts or studs 4, 5 is a top housing 6. The top housing forms an upper distribution chamber 7 into which is directed an inlet feed pipe 8. The inlet feed pipe is provided with an O-ring seal 9 and a flange

- 11 -

10. A locking ring 11 is bolted to the upper face 12 of the top housing 6 to trap the flange 10 to hold the inlet feed pipe on the top housing. Suitable bolts or studs 13, 14 are provided to bolt the ring 11 to the top housing 6.

5 Bolted to the underside of the top housing 6 is a bottom housing 20. A series of bolts 21, 22 are used to bolt the top and bottom housing together and an annular spacer 23 forms a positive stop to locate the top and bottom housings together at a predefined distance.

10 The bottom housing 20 has an inwardly directed flange portion 24 which has an annular upwardly directed surface 25. The upper housing 6 has an annular downwardly directed horizontal clamping face 26.

Clamped between the faces 25 and 26 is a spinnerette, a  
15 breaker plate and filter assembly. The spinnerette, shown in perspective view in Figure 3, essentially comprises a rectangular member in plan view, having a top hat cross section and comprising an upwardly directed peripheral wall generally indicated by 28 incorporating an integral outwardly  
20 directed flange portion 29. The spinnerette incorporates a plurality of aperture plates 30, 31, 32 which contain the holes through which the solution of cellulose in amine oxide, 33 is spun or extruded to form filaments 34.

Located on the upper surface of the flange 29 is a gasket  
25 35. Located on top of the gasket 35 is a breaker plate 36 which essentially comprises an apertured plate used to support a filter element 37. The filter element 37 is formed of sintered metal, and if the sintered metal has a fine pore size, the pressure drop across the filter can, in use, rupture  
30 the filter. The breaker plate 36, therefore, supports the filter in use. A pair of gaskets 38, 39 on either side of the filter completes the assembly located between the upwardly directed face 25 of the bottom housing and the downwardly directed face 26 of the top housing. By clamping the assembly

- 12 -

together with the bolts 21, 22, the spinnerette, breaker plate and filter are held positively in position.

Located beneath the bottom housing 20 is an annular thermally insulating ring 40 which is generally rectangular in plan shape. The annular insulating ring extends around the complete periphery of the wall 28, which wall 28 extends below the lower face 41 of the bottom housing 20. On one long side of the spinnerette, there is provided an integral extension portion 42 of the insulating ring 40 which extends below the long wall portion 43 of the peripheral wall 28. On the other long wall portion 41 of the peripheral wall 28 the insulating ring 40 does not have the integral extension portion 42, but the lower face 44 of that portion of the ring 40 is in the same plane as the face 46 of the portion 41 of the peripheral wall 28 of the spinnerette.

As is more easily seen in Figure 2, the insulating ring 40 which is secured to the underside of the bottom housing 20 by screws (not shown) has the integral extension portions 50, 51 extending over the lower faces of the portions 52, 53 of the shorter lengths of the peripheral wall 28 of the spinnerette.

Referring to Figure 3 this shows in perspective the spinnerette incorporated into the jet assembly. The spinnerette, generally indicated at 60, has an outer flange 29 integral with the wall 28. The rectangular nature of the spinnerette can clearly be seen from the perspective view in Figure 3. The minor axis of the spinnerette is shown in the sectional view of Figure 1 and the major axis is shown in sectional view in Figure 2. Welded into the bottom of the spinnerette are six aperture plates 61 of which three of the plates 30, 31, 32 can be seen in sectional view in Figure 1. These plates contain the actual holes through which the cellulose solution is extruded. The holes can have a diameter in the range  $25\mu$  to  $200\mu$  and be spaced by 0.5 to 3mm in a centre-to-centre measurement. The spinnerette has an

- 13 -

underside in a single plane and is capable of withstanding the high extrusion pressures experienced in spinning a hot cellulose solution in amine oxide. Each plate can contain between 500 and 10,000 holes, i.e. up to 40,000 holes for jets 5 with four plates. Up to 100,000 holes can be used.

Figure 4, is an underneath view of the spinnerette showing the location of the insulating annular member 40. It can be seen that the insulating layer, typically formed of a resin impregnated fabric material such as Tufnol (trade mark) 10 extends below the lower portion of the peripheral wall 28 on three sides of the spinnerette. Thus, seen from below, on sides 62, 63 and 64, the lower portion of the wall 28 is obscured by the extension portions in the insulating layer shown as 42, 50 and 51 in Figures 1 and 2. However, on the 15 fourth side, side 65, the lower portion 66 of the wall 28 of the spinnerette 60 is not insulated and is, therefore exposed. The insulating annulus, therefore, is effectively surrounding the spinnerette completely and extends on three sides beneath the peripheral wall of the wall of the spinnerette.

20 It will be noted that the breaker plate 36 has tapered holes 67 which enhance the flow of viscous cellulose solution through the jet assembly whilst providing a good support for the filter 37. In turn the breaker plate 36 is supported by the upper edges of the internal bracing members or spars 68, 25 69, 70. The upper edges of the internal bracing members or spars may be displaced from the centre line of the members or spars so that the entrance area above each aperture plate is equal.

30 The facings 25, 26 of the housing and/or the breaker plate 36 may be provided with small recesses such as recess 80 (see Figure 2) so as to permit the gasket to be extruded into the recess to enhance sealing when the bolts holding the top and the bottom housing together are tightened. An O-ring 35 84 may be provided between the top and bottom housing to act as a second seal in the event of failure of the main seals

- 14 -

between the top and bottom housing and the breaker plate and filter assembly.

A spinnerette as employed in the invention is, therefore, capable of handling highly viscous high pressure cellulose solution in which typically the pressure of the solution upstream of the filter may be in the range 50 to 200 bar and the pressure at the inside of the die face may be in the range 20 to 100 bar. The filter itself contributes to a significant amount of pressure drop through the system whilst in operation.

The assembly of the invention also provides a suitable heat path whereby the temperature of the dope in the spinning cell can be maintained close to the ideal temperature for spinning for extrusion purposes. The bottom housing 20 is in firm positive contact with the spinnerette through its annular upwardly directed face 25. The bolts or set screws 21, 22 ensure a firm positive contact. Similarly, the bolts 4, 5 positively ensure that the bottom housing 20 is held tightly to the frame member 22 via its downwardly directed face 81 formed on an outwardly directed flange portion 82. The face 81 is in positive contact with the upwardly directed face 83 of the housing 2.

By providing a heating element in the form of a heating tube 3 directly below the face 83 there is a direct flow path for heat from the heating medium in the bore 3 into the spinnerette. It can be seen that heat can flow through the faces 83, 81 which, as mentioned above, are held in positive contact by the set screws 4, 5. Heat can then flow through the bottom housing 20 via the face 25 and flange 29 into the spinnerette wall 28.

It will readily be appreciated that assemblies of the type illustrated in the accompanying drawings are normally assembled in an ambient temperature workshop. Thus typically the top and bottom housing, the spinnerette, the breaker plate



- 15 -

and filter plate assembly will be bolted up at ambient temperature by tightening the screws 21, 22. To enable the spinnerette to be inserted into the bottom housing 20 there needs to be a sufficient gap between the peripheral wall 28 and the interior hole of the bottom housing 20 which permits the spinnerette to be inserted and removed. It will also be appreciated that in use the assembly is heated to typically 100°C. The combination of heating and internal pressure means that there will be an unregulated expansion of the assembly.

10 All of this means that it is not possible to rely upon a direct heat transfer sideways from the lower portion of the bottom housing directly horizontally into the side of the peripheral wall 28.

Similar constraints apply to the direct horizontal transfer of heat into the outer side wall of the bottom housing 20 directly from the heated lower portion of the frame 2. However, by providing for a positive clamped face-to-face surface such as surface 81, 83, a positive route for the transfer of heat from the medium within bore 3 to the spinnerette is provided. Any suitable heating medium such as hot water, steam or heated oil can be passed through the bore 3.

The provision of the lower thermal insulation 40 whilst not needed from a safety-to-personnel view point ensures that the heat from the hot cellulose solution itself is passed into the jet assembly from the bore 3 and does not escape through the lower face of the bottom housing.

It will readily be appreciated that the components of the spinning cell should be manufactured from material capable of withstanding any solvent solution passed through it. Thus, for example, the spinnerette may be made from stainless steel and the housings may be made from stainless steel or castings of cast iron as appropriate. The gaskets may be formed of PTFE.

- 16 -

Without prejudice to the present invention it is believed that the cross-draught tends to evaporate some of the water contained in the cellulose NMMO water solution so as to form a skin on the filaments as they emerge from the spinnerette.

5 The combination of the cooling effect of the cross-draught and the evaporation of moisture from the filaments cools the filaments, thus forming a skin which stabilises the filaments prior to their entry into the spin bath. This means that very large numbers of filaments can be produced at a single time.

10 At the bottom end of the spinning cells, the holes 103 are each provided with gaiters as is illustrated in more detail in Figure 8. The tow 130 of filaments passes through the hole 103 into a resilient gaiter 131 which is located at its upper end in firm and liquid-tight contact with the wall

15 in which the hole 103 is provided. The gaiter 131 has an aperture at its lower end slightly smaller in diameter than the tow 130. The gaiter is formed of neoprene rubber and the tow 130 stretches the rubber slightly so as to form a form contact with the tow as it passes through the gaiter. The

20 gaiter thus restricts the excess flow of liquor out of the bottom of the spinning cell.

The tow subsequently passes underneath a godet and then upwardly for washing and further processing. Below the godet there may be provided a drip tray to catch spin bath liquor

25 entrained in the tow and passing through the gaitered hole 103.

The flow of spin bath liquid in the upper portion of the spinning cell will now be described more clearly with reference to Figures 9 and 10. Figure 9 shows a perspective

30 plan view of an empty upper portion of a spinning cell. The spinning cell effectively comprises a liquid-tight vessel defined by side walls 135 and 136 and by end walls 137 and 138. The side walls 135 and 136 are continuous steel side walls, whereas the end walls 137 and 138 are provided with

35 doors 139 and 140 as described more fully below.

- 17 -

Outside of the liquid-tight spinning cell defined by the walls 135 to 138, there is an external framework defined by side walls 141 and 142 and end walls 143 and 144. It can be seen that the end walls 143 and 144 are provided with U-shaped cut outs generally indicated by 145 and 146. The upper edges of the side walls 135 and 136 are slightly below the upper edges of the end walls in particular that portion of the end walls defined by doors 139 and 140. The doors may be formed of metal or may be formed of glass or a clear plastics material. The doors are mounted in the side walls so that they may be conveniently opened. The doors may, for example, be hinged at their lower edges and held in closed position by means of side bolts or the doors may be bolted around three sides to the side walls of the cell.

15 In use, a slight excess of liquid is pumped into the spinning cell and the excess liquid overflows the upper sides of the edges 135 and 136 to form an upper surface of liquid in the cell. If desired the upper edges may be serrated.

On the suck side of the cell, there is preferably provided a liquid trap. This is shown more clearly in Figure 10 but it essentially comprises a channel formed between an angled wall 147 and the upper portion of the side wall 135. The suck nozzle 148 has a dependent strip 149 which extends below the upper surface of the channel 147. Excess liquid then flows over the upper edge 150 into the channel 151 to fill the channel and overflow as at 152 into a gutter 153. Excess liquid flows out of a pipe 154 from the gutter 153 to be recycled as required. The effect of the combination of the liquid in the channel 151 together with the dependent strip 149 is to form a gas-tight seal to prevent the suction nozzle 148 sucking air up along the side of the cell between the walls 141 and 135.

By providing the hole 103 at the bottom of the spin bath cell as described above, the initial lacing up of the tow to commence preparation of the production of lyocell fibres is

- 18 -

considerably eased. The process for commencing production, therefore, simply comprises spinning a small quantity of fibres into the cell and then hooking the fibres through the hole in the bottom to pull the tow downwardly around the lower  
5 godet or roller (not shown) and then thread the tow onwardly through the following fibre washing and fibre drying sections (not shown).

Because of the narrow gap between the upper end of the spinning cell and the lower regions of the jet assembly,  
10 lacing up of the tow is considerably eased by the provisions of the doors 139 and 140. To lace up the cell at the commencement of a spinning operation, the doors 139 and 140 are opened - the spin bath liquid from the cell then falling into the surrounding catchment troughs. Spinning is then  
15 commenced and the spun fibres can be manipulated and pushed through the hole at the bottom of the cell. Once the cell has been laced up, the door 139, 140 can be closed, the cell refilled and operation can then be continued automatically.

If required, plain water can be used in the spin bath for  
20 starting purposes. This water tends to froth less than aqueous amine oxide mixtures and eases start-up of the cell. The provision of the doors 139, 140 also enables ready access to the interior of the spin bath and to the edges of the suck nozzle. This enables small quantities of crystalline growth  
25 which appear on the cell during operation to be removed. It is believed that these crystalline growths arise from the slight evaporation of amine oxide.

It will be appreciated that a large number of cells may be aligned in a side-by-side relationship and the bottom of  
30 each cell can readily be assessed by an operator. If on the other hand the fibres emerge through the upper surface of the spin bath, the lacing up of the system is very much more complicated and involves an operator trying to work below the surface of the spin bath to collect the fibres in a tow from  
35 below the surface of the spin bath. Additionally, when large

- 19 -

numbers of cells are placed in side-by-side relationship it becomes difficult to access the top of the cells, particularly if the air gap is very small and the cells are narrow. It can be seen that by utilising a narrow and little larger than the 5 wedge of tow passing through the spin bath.

- 20 -

CLAIMS

1. A method for the production of cellulose filaments from a solution of cellulose in an organic solvent, characterised in that it comprises the steps of extruding the solution  
5 through a die (60) having a plurality of holes to form a plurality of strands, passing the strands (125) across a gaseous gap into a water-containing spin bath (101, 115) to form the filaments and providing a forced flow of gas through the gap parallel to the upper surface (116) of the water in  
10 the spin bath (101, 115).
2. A method as claimed in claim 1, characterised in that the gas is sucked across the gap.
3. A method as claimed in Claim 1 or 2, characterised in that the die (60) has between 500 and 100,000 holes,  
15 preferably between 1,000 and 15,000 holes and further preferably between 2,000 and 10,000 holes.
4. A method as claimed in Claim 1, 2 or 3, characterised in that the solution of cellulose is maintained at a temperature in the range 100°C to 125°C.
- 20 5. A method as claimed in any one of claims 1 to 4, in which the gas is both sucked and blown across the gap.
6. A method as claimed in Claim 1, 2, 3, 4 or 5, characterised in that the gas is air.
7. A method as claimed in Claim 6, characterised in that the  
25 air has a dew point of 10°C or below.
8. A method as claimed in Claim 6 or 7, characterised in that the air is at a temperature between 0°C and 50°C.
9. A method as claimed in any one of Claims 1 to 8, in which the gap is between 0.5cm and 25cm in height.

- 21 -

10. A spinning cell for the coagulation of filaments formed from a solution of cellulose in an organic solvent, characterised in that the cell includes a spin bath (101, 115) for the leaching of the solvent from the filaments (34) and  
5 a gap above the spin bath (101, 115), the gap being defined at the lower side by the surface of the spin bath (101, 115) and at the upper side by a spinnerette (60) from which the filaments (34) emerge, and means to provide a flow of gas across the gap.
- 10 11. A spinning cell as claimed in claim 10, characterised in that said means comprises a suck nozzle (122, 148) having an entrance on one side of the gap.
12. A spinning cell according to Claim 11, characterised in that a blow nozzle (121) is positioned to have an exit on the  
15 opposite side of the gap to the entrance to the suck nozzle (122, 148).
13. A spinning cell according to Claim 12, characterised in that the suck nozzle (122, 148) has a greater cross-sectional area at its entrance than the blow nozzle (121) has at its  
20 exit.
14. A spinning cell according to any one of Claims 10 to 13, characterised in that baffle means (108, 109, 110, 111, 112) are located within the spin bath to restrict the flow of  
25 currents of liquid within the spin bath and to calm the surface of the liquid.
15. A spinning cell according to any one of Claims 10 to 14, characterised by an aperture (108) at the lower end of the spin bath through which coagulated filaments emerge in the  
30 form of a tow (130), and a gaiter (131) of flexible resilient material having an orifice which in the unrestrained condition is slightly smaller in cross-sectional area than the tow (130), the gaiter being sealingly secured at its upper end around the aperture (103) at the lower end of the spin bath

- 22 -

(101, 115), the tow passing, in use, through the orifice and thereby expanding the cross-sectional area of the orifice.

16. A spinning cell as claimed in any one of Claims 10 to 15, characterised in that the cell (101, 115) is rectangular in shape with the blow nozzle (122, 148) on one of the longer sides.

17. A spinning cell as claimed in Claim 16, characterised in that there is an access door (139, 140) in at least one shorter side of the cell.

18. A spinning cell as claimed in any one of Claims 10 to 17, characterised in that the upper edge (150) of the cell on the suck side (148) acts as a weir to define the level of liquid in the cell.

19. A spinning cell as claimed in Claim 18, characterised in that there is a drainage passage (153) on the outside of the wall having the weir.

20. A spinning cell as claimed in Claim 19, characterised in that the drainage passage (153) includes a liquid trap (149, 151) to prevent air being sucked up the drainage passage.

21. A spinning cell as claimed in any one of Claims 10 to 20, characterised in that there is provided a thermally insulating layer (40) beneath the side walls of the spinnerette (60) on at least the blow side.

22. A method for the production of cellulose filaments from a solution of cellulose in an organic solvent, characterised in that the solution is extruded through a die (60) having a plurality of holes to form a plurality of filaments (34), the filaments are passed through a water-containing spin bath (101, 115) to leach solvent from the filaments and the tow of filaments (130) is passed through a hole (103) at the lower end of the spin bath (101, 115), the hole (103) being provided



- 23 -

with a resilient periphery to resiliently contact the tow.

23. A method according to Claim 22, characterised in that the hole (103) is provided with a resilient gaiter (131) to provide the resilient periphery to contact the tow.

5 24. A method according to Claim 23, characterised in that the gaiter (131) has an orifice at its lower end slightly smaller in diameter than the tow (130).

25. A method according to Claim 22, 23 or 24, characterised in that the filaments (34) are passed through a gap between  
10 the die (60) and the spin bath (101, 115) and a forced flow of gas is provided through the gap parallel to the upper surface of the liquid in the spin bath.

26. A spinning cell for the coagulation of cellulose filaments formed from a solution of cellulose in an organic  
15 solvent, characterised in that the cell has a spin bath (101, 115) for the leaching of the solvent from a tow (130) of the filaments, the lower end of the spin bath (101, 115) having a hole (103) through which the tow (130) can be passed, the hole (103) being provided with a resilient periphery to  
20 resiliently contact the tow (130).

27. A spinning cell according to Claim 26, characterised in that the resilient periphery is provided by a resilient gaiter (131).

28. A spinning cell according to Claim 27, characterised in  
25 that the resilient gaiter (131) is sealingly secured at its upper end around the hole (103) and has an aperture at its lower end slightly smaller in diameter than the tow of filaments (130).

29. A spinning cell according to Claim 26, 27, or 28,  
30 characterised in that a gap is provided above the spin bath (101, 115) and is defined between the upper surface of the spin

- 24 -

bath and the lower surface of a die (60) through which the filaments (34) are formed.

30. A spinning cell according to Claim 29, characterised in that means (121, 122) are provided to provide a forced flow  
5 of gas through the gap parallel to the upper surface of the spin bath (101, 115).

31. A method for the production of cellulose filaments from a solution of cellulose in an organic solvent, characterised in that the solution is extruded through a die (60) having a  
10 plurality of holes to form a plurality of filaments (34), the filaments are passed as a tow (130) through a water-containing spin bath (101, 115) to leach solvent from the filaments, and baffles (108, 109, 110, 111, 112) are provided in the spin bath to reduce turbulence.

15 32. A method according to Claim 31, characterised in that the cross-sectional area of the tow (130) is reduced as it travels towards the outlet of the spin bath (101, 115).

33. A method according to Claim 31 or 32, characterised in that the baffles (108, 109, 110, 111, 112) are porous.

20 34. A method according to Claim 31, 32 or 33, characterised in that the baffles (108, 109, 110, 111, 112) are provided at a plurality of levels in the spin bath (101, 115).

35. A method according to any one of Claims 31 to 34, characterised in that the filaments (34) are passed through  
25 a gap between the die (60) and the spin bath (101, 115) and a forced flow of gas is provided through the gap parallel to the upper surface of the liquid in the spin bath.

36. A spinning cell for the coagulation of cellulose filaments formed from a solution of cellulose in an organic  
30 solvent, characterised in that the cell has a spin bath (101, 115) for the leaching of the solvent from a tow (130) of the

- 25 -

filaments as it is passed through the spin bath (101, 115), the spin bath having baffles (108, 109, 110, 111, 112) to reduce turbulence.

37. A spinning cell according to Claim 36, characterised in  
5 that the baffles (108, 109, 110, 111, 112) are porous.

38. A spinning cell according to Claim 36 or 37, characterised in that the baffles (108, 109, 110, 111, 112) are perforated plates.

39. A spinning cell according to Claim 36, 37 or 38,  
10 characterised in that the baffles (108, 109, 110, 111, 112) are positioned at a plurality of levels in the spin bath (101, 115).

40. A spinning cell according to any one of Claims 36 to 39,  
characterised in that the baffles (108, 109, 110, 111, 112)  
15 are positioned within the upper region of the spin bath (101, 115).

41. A spinning cell according to any one of Claims 36 to 40,  
characterised in that the baffles (108, 109, 110, 111, 112)  
are shaped to be positioned close to the moving surfaces of  
20 the tow or tows (130) of filaments passing through the spin bath (108, 109, 110, 111, 112).

42. A spinning cell according to any one of Claims 36 to 41,  
characterised in that a gap is provided above the spin bath  
(101, 115) and is defined between the upper surface of the  
25 spin bath and the lower surface of a die (60) through which the filaments (34) are formed.

43. A spinning cell according to Claim 42, characterised in  
that means (121, 122) are provided to provide a forced flow  
of gas through the gap parallel to the upper surface of the  
30 spin bath (101, 115).

- 26 -

44. A method for the production of cellulose filaments from a solution of cellulose in an organic solvent substantially as hereinbefore described with reference to and as shown in the accompanying drawings.
- 5 45. A spinning cell for the coagulation of filaments formed from a solution of cellulose in an organic solvent substantially as hereinbefore described with reference to and as shown in the accompanying drawings.
46. Cellulose filaments produced by the method or with the  
10 apparatus claimed in any one of claims 1 to 45.

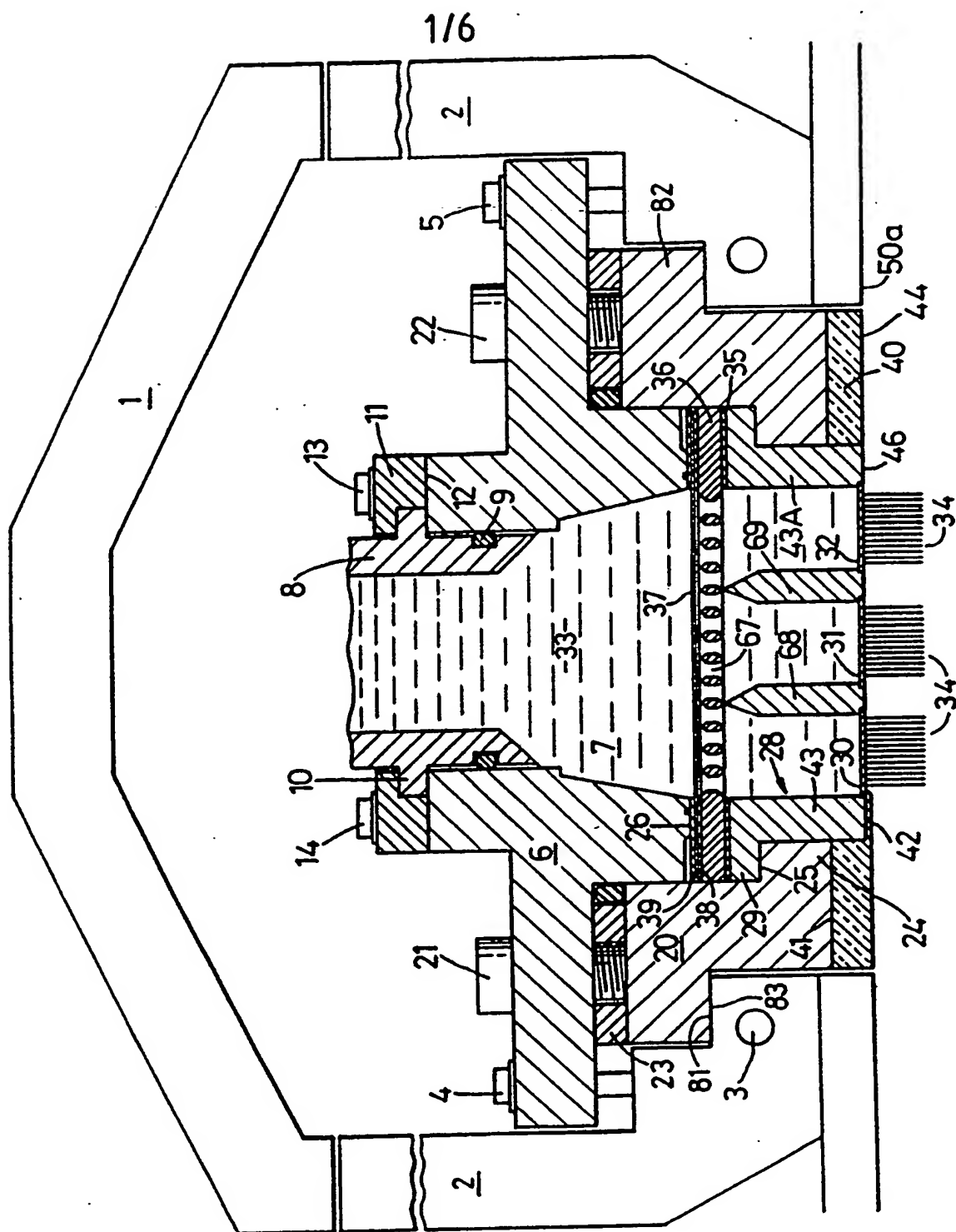


Fig.1

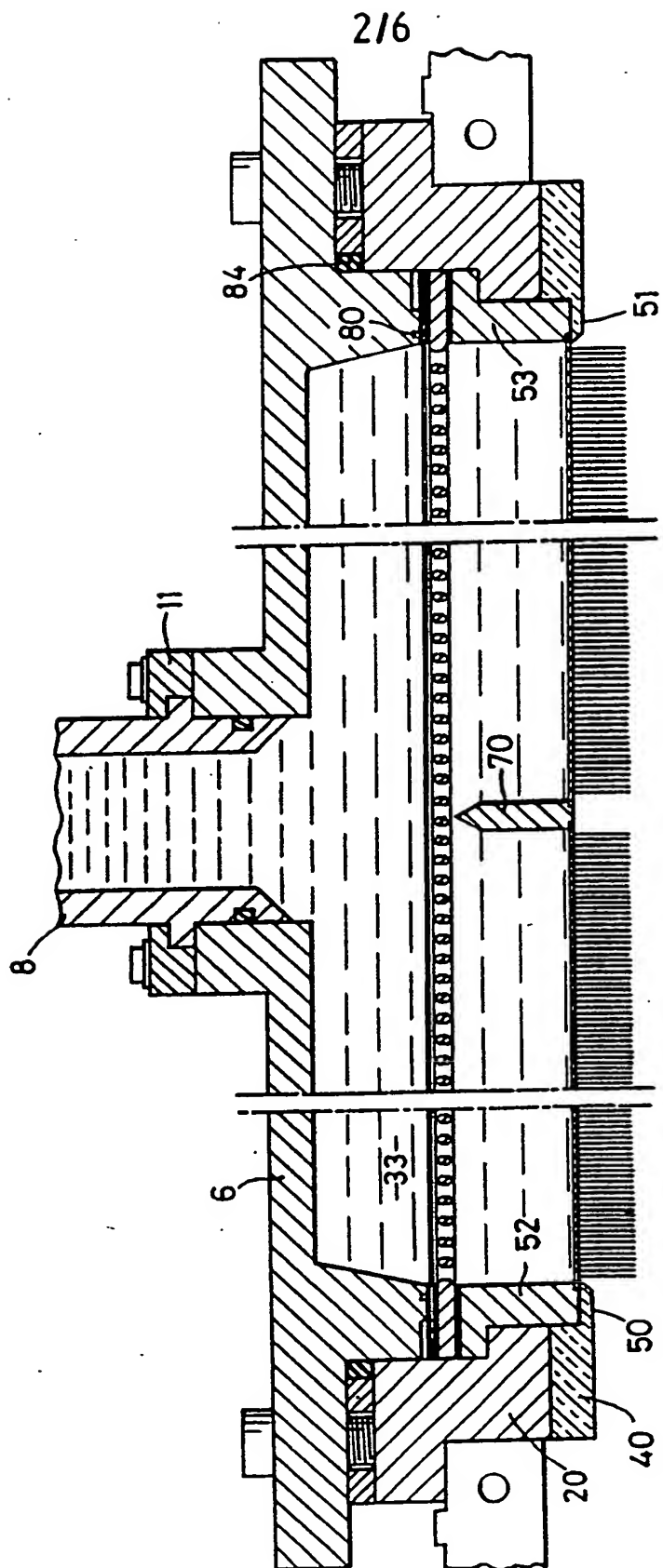


Fig. 2

3/6

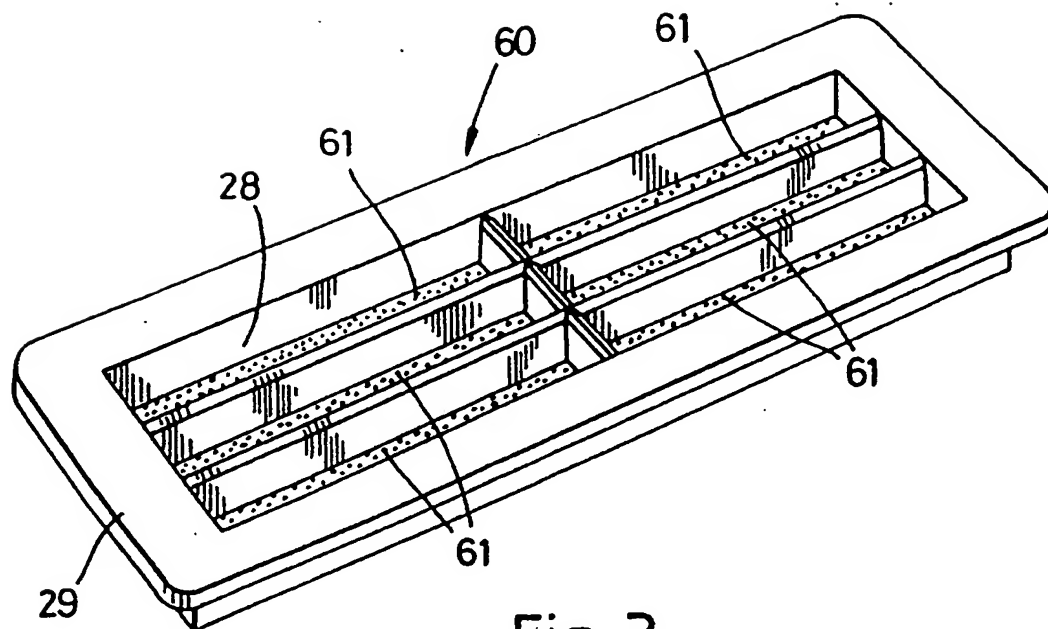


Fig. 3

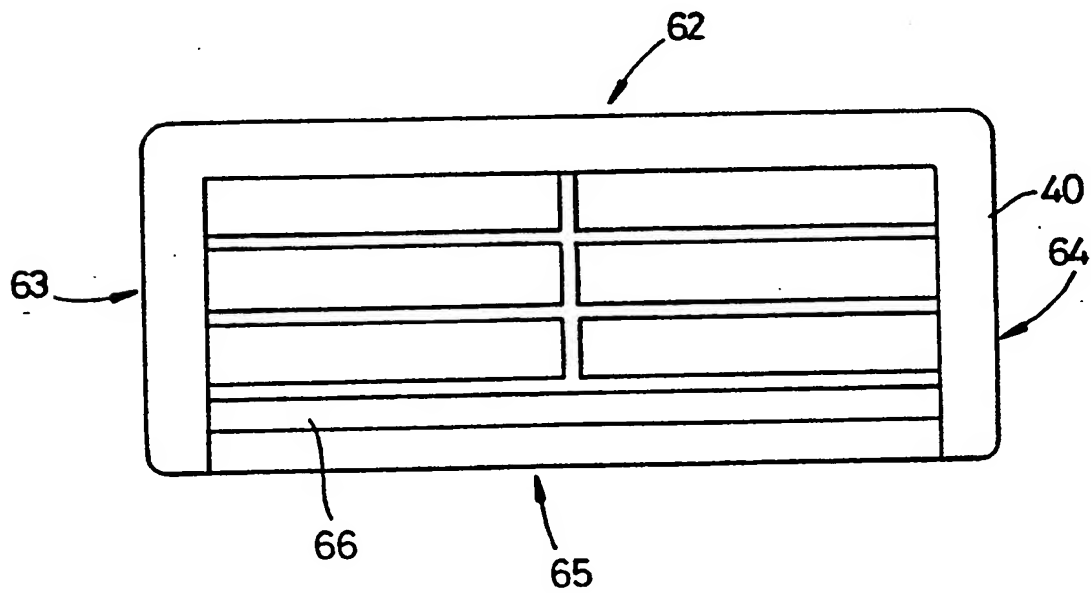
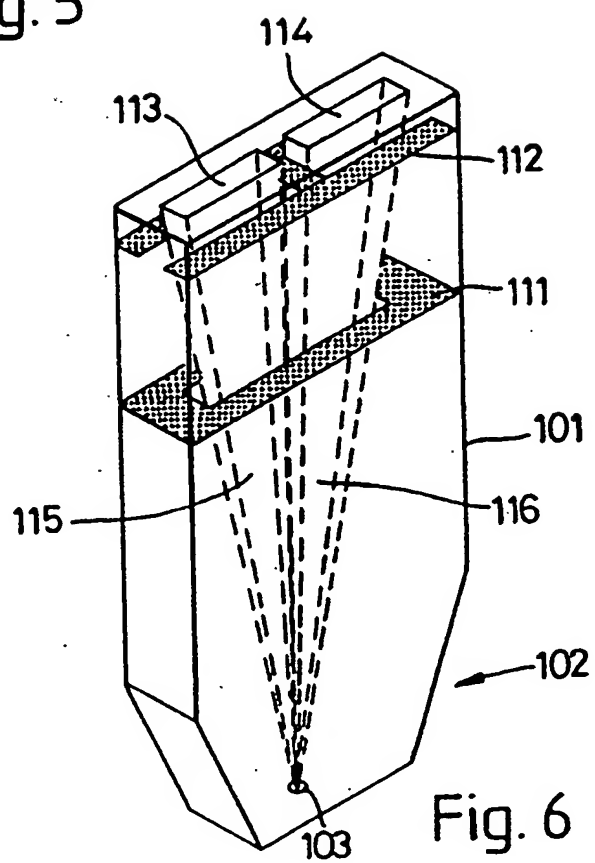
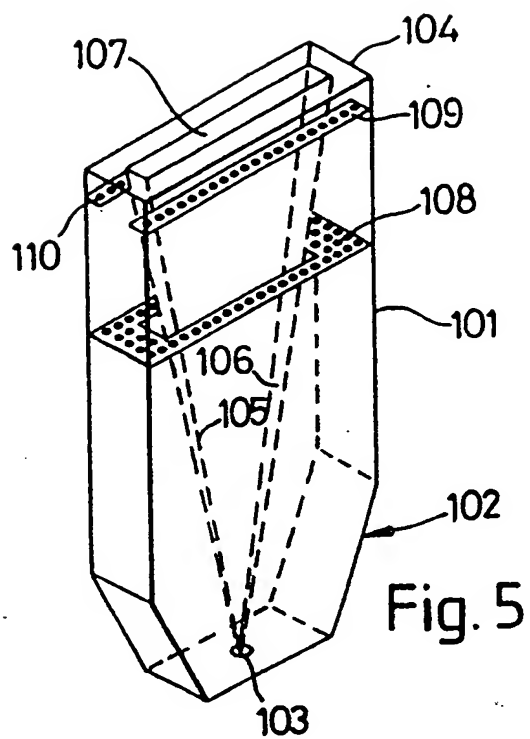
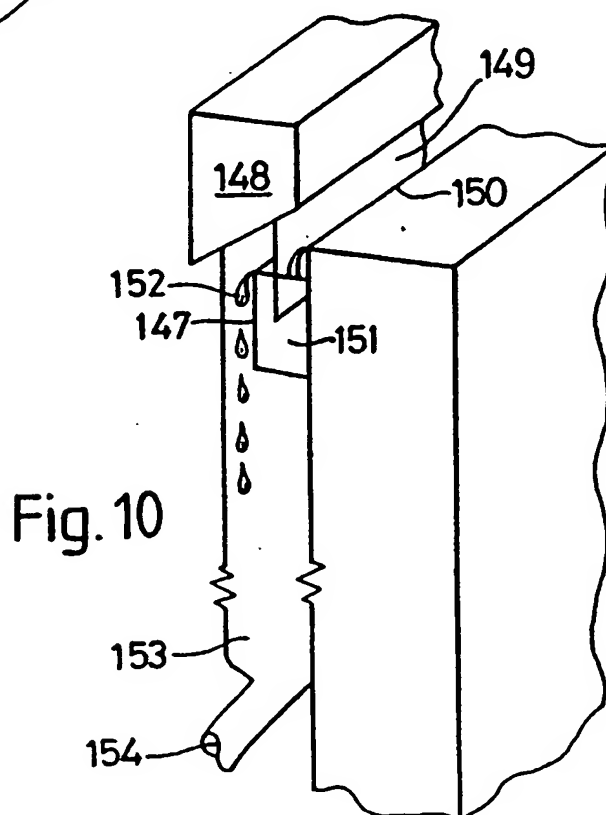
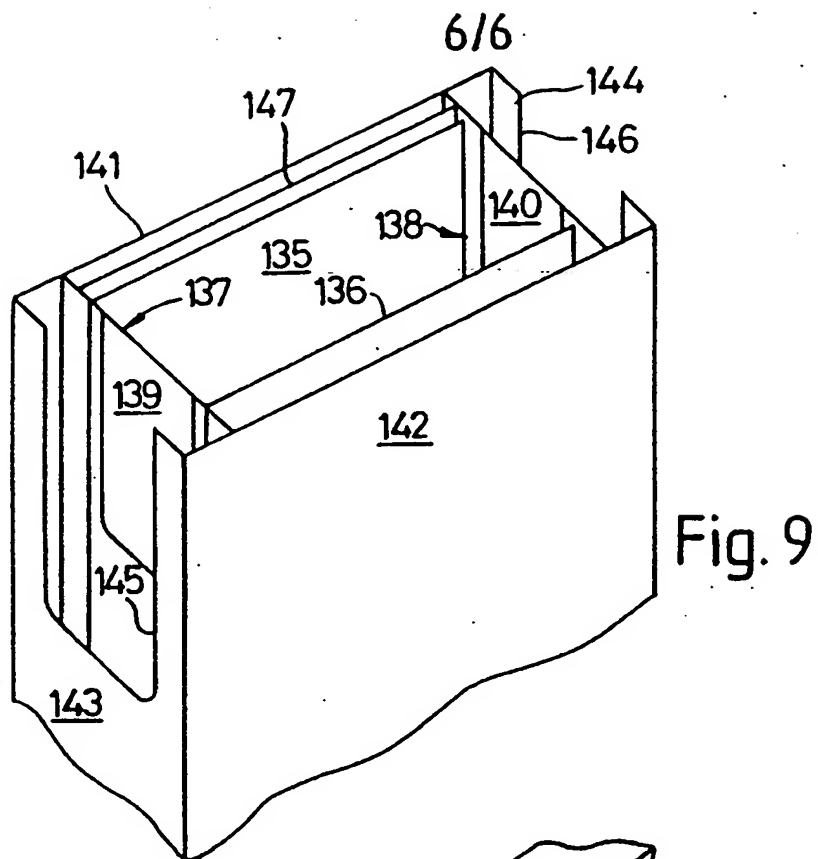


Fig. 4

4/6







## INTERNATIONAL SEARCH REPORT

**Intr      onal Application No**

**PCT/GB 94/01107**

### A. CLASSIFICATION OF SUBJECT MATTER

A. CLASSIFICATION OF SUBJECT MATTER  
IPC 5 D01F2/00 D01D5/06 D01D5/088

According to International Patent Classification (IPC) or to both national classification and IPC

### B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 5      D01F      D01D

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

### C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
P,X	WO,A,93 19230 (LENZING AKTIENGESELLSCHAFT) 30 September 1993 see the whole document ---	1-10,25, 35,44-46
A	PATENT ABSTRACTS OF JAPAN vol. 17, no. 349 (C-1078)2 July 1993 & JP,A,05 044 104 (UNITIKA LTD) 23 February 1993 see abstract ---	2,5,11, 30,43
A	US,A,3 905 381 (RICHARD D. MEYER) 16 September 1975  see the whole document ---	14, 31-34, 36-42
	---	
	-/--	

**X** Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

\* Special categories of cited documents :

- \* "A" document defining the general state of the art which is not considered to be of particular relevance
- \* "E" earlier document but published on or after the international filing date
- \* "L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
- \* "O" document referring to an oral disclosure, use, exhibition or other means
- \* "P" document published prior to the international filing date but later than the priority date claimed

T later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

- \*X\* document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
- \*Y\* document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.
- \*A\* document member of the same patent family

Date of the actual completion of the international search

**23 September 1994**

Date of mailing of the international search report

03.10.94

Name and mailing address of the ISA

European Patent Office, P.B. 5818 Patentlaan 2  
NL - 2280 HV Rijswijk  
Tel. (+ 31-70) 340-2040, Tx. 31 651 epo nl,  
Fax (+ 31-70) 340-3016

Authorized officer

Tarrida Torrell, J

# INTERNATIONAL SEARCH REPORT

Int. Patent Application No  
PCT/GB 94/01107

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT		
Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US,A,3 221 088 (EMMETT V. MARTIN) 30 November 1965  see column 3, line 34 - line 55; figure 2 ---	15, 22-24, 26-30
A	DE,C,715 504 (CARL HAMEL AG) 23 December 1941 see the whole document ---	18-20
A	GB,A,1 298 413 (IMPERIAL CHEMICAL INDUSTRIES LIMITED) 6 December 1972 see the whole document -----	21

# INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No  
PCT/GB 94/01107

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
WO-A-9319230	30-09-93	AU-B- 3621193 CA-A- 2102809 CZ-A- 9302364 EP-A- 0584318 HU-A- 65897	21-10-93 18-09-93 13-04-94 02-03-94 28-07-94
US-A-3905381	16-09-75	US-A- 4037288	26-07-77
US-A-3221088		NONE	
DE-C-715504		NONE	
GB-A-1298413	06-12-72	AU-B- 456152 AU-A- 3186271 DE-A- 2138201 FR-A- 2103925 NL-A- 7110508 US-A- 3752874	12-12-74 01-02-73 10-02-72 14-04-72 08-02-72 14-08-73

**This Page is Inserted by IFW Indexing and Scanning  
Operations and is not part of the Official Record**

**BEST AVAILABLE IMAGES**

Defective images within this document are accurate representations of the original documents submitted by the applicant.

Defects in the images include but are not limited to the items checked:

☐ BLACK BORDERS

☐ IMAGE CUT OFF AT TOP, BOTTOM OR SIDES

☐ FADED TEXT OR DRAWING

☐ BLURRED OR ILLEGIBLE TEXT OR DRAWING

☐ SKEWED/SLANTED IMAGES

☐ COLOR OR BLACK AND WHITE PHOTOGRAPHS

☐ GRAY SCALE DOCUMENTS

☒ LINES OR MARKS ON ORIGINAL DOCUMENT

☐ REFERENCE(S) OR EXHIBIT(S) SUBMITTED ARE POOR QUALITY

☐ OTHER: \_\_\_\_\_

**IMAGES ARE BEST AVAILABLE COPY.**

**As rescanning these documents will not correct the image problems checked, please do not report these problems to the IFW Image Problem Mailbox.**